Method of Test for

DETERMINING ALKALI CONTENT OF FLY ASH BY FLAME PHOTOMETER

DOTD Designation 531-11

I. Scope

Use this method to determine Na_2O and K_2O content of fly ash for calculation of equivalent percentage of sodium oxide by weight.

II. Apparatus

- A. Fly Ash Worksheet (Figure 1)
- B. **Analytical Balance** 120 g capacity accurate to +/-0.0001 g (Figure 2)
- C. Flame photometer (Figure 3)
- D. Hot plate (Figure 4)
- E. Teflon Beaker 100 ml (Figure 5)
- F. Stirring rod
- G. 200 ml volumetric flask
- H. 75 mm glass funnel
- Medium texture filter paper –
 Whatman No. 40 Filter Paper or equivalent
- J. Spatula
- K. Weighing paper or weighing boat

III. Reagents

- A. Diluted HCL (1 part concentrated HCL specific gravity of 1.19 ACS certified to 4 parts of Deionized Water)
- B. Deionized water

IV. Health Precautions

Proper precautions are to be taken whenever hot materials or equipment must be handled. Use container holder or thermal gloves while handling hot containers. Wear eye protection while stirring and weighing materials. Perform the preparation of sample under vent hood to prevent exposure to fumes.

V. Sample

Obtain sample according to DOTD S102 sampling procedure as stated in Material sampling Manual.

VI. Procedure

- A. Weigh 0.25 g of fly ash and transfer to Teflon beaker using the weighing paper and spatula.
- B. Add 40 ml of diluted HCL.
- C. Use stirring rod to mix (and if necessary, grind) test specimen.
- D. Digest test specimen at a temperature just below boiling on hot plate until the test specimen is completely digested.
- E. Filter solution with test specimen through Whatman 40 filter paper (or equivalent) via 75 mm glass funnel into 200 ml volumetric flask.
- F. Rinse the beaker with 10 ml of hot deionized water (approximately 80°C -100°C).
- G. Wash filter paper containing test specimen thoroughly with a stream of 10 to 15 ml hot deionized water (85°C -95°C).
- H. Dilute filtrate to 200 ml with deionized water up to the mark on the volumetric flask. Then, mix thoroughly.
- I. Determine Na₂O and K₂O content utilizing flame photometer in accordance with the flame photometer manufacturer's operating procedure.

VII. Calculation

A. Calculate the results as percent by weight of the original fly ash sample.

B. The equivalent percentage of sodium oxide (Na₂O), calculated as follows:

Equivalent Na_2O , % = Na_2O , % + (0.658 x K_2O , %)

example:

$$Na_2O = 1.42\%$$

 $K_2O = 0.17\%$

Equivalent $Na_2O\% = 1.42 + (0.658 \times 0.17)$

Equivalent $Na_2O\% = 1.42 + 0.11$

Equivalent Na₂O% = 1.53

Equivalent $Na_2O\% = 1.5$

VIII. Report

The data shall be reported to the nearest tenth of a percent (0.1%) on the Fly Ash worksheet (Figure 1).

Example: 1.53 is reported as 1.5

IX. Normal Testing Time

Normal test reporting time is three (3) days.

FLY ASH, MATERIAL CODES 297 AND 589 CHEMICAL PROPERTIES TOTAL OXIDES (ASTM C311) 58.0 SIL CON DIOXIDE 34.1 AMMONIUM HYDROXIDE GROU? 23.9 SULFUR TRIOXIDE (ASTM C311) 2.2 MAGNESIUM CXIDE (ASTM C311) 6.8 CALCIUM OXIDE (ASTM C311) 26.1 1.2 LOSS ON IGNITION, % (ASTM C311) ALKALIES, % (ASTM C114) AVAILABLE ALKALIES, % (ASTM C311) 0.4 MOISTURE CONTENT, % (ASTM 0311) TESTED BY: DATE: 11/15/2010 CHECKED BY: DATE: 11/15/2010 DATE: 11/15/2010 Sample 22-743705

Figure 1
Fly Ash Worksheet



Figure 2
Analytical Balance

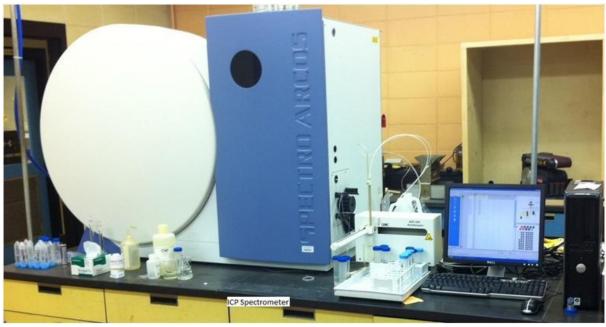


Figure 3
Flame Photometer



Figure 4 Hot Plate



Figure 5
Other Apparatuses